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Treatability Test Report

FOTH INFRASTRUCTURE and ENVIRONMENT, LLC

2723 S. Ridge Road
Green Bay, WI 54307

Treatment of Simulated Mine Wastewater
Kennecott Minerals Company

Report No. JWM0726

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SECTION I BACKGROUND

Siemens Water Technologies was contracted by Foth Infrastructure and Environment to evaluate the effectiveness of using a “minimum-liquid discharge” system to treat wastewater generated at a proposed mine in Michigan.

The system consists of two major subsystems: the primary reverse osmosis treatment system and the concentrate reverse osmosis treatment system. The primary reverse osmosis (RO) subsystem includes the following processes:

- Chemical softening / Clarification
- Sand filtration
- Reverse osmosis (2 pass)

The concentrate reverse osmosis treatment subsystem consists of the following processes:

- Chemical softening / Microfiltration
- Cation exchange resin
- Reverse osmosis
- Boron specific ion exchange

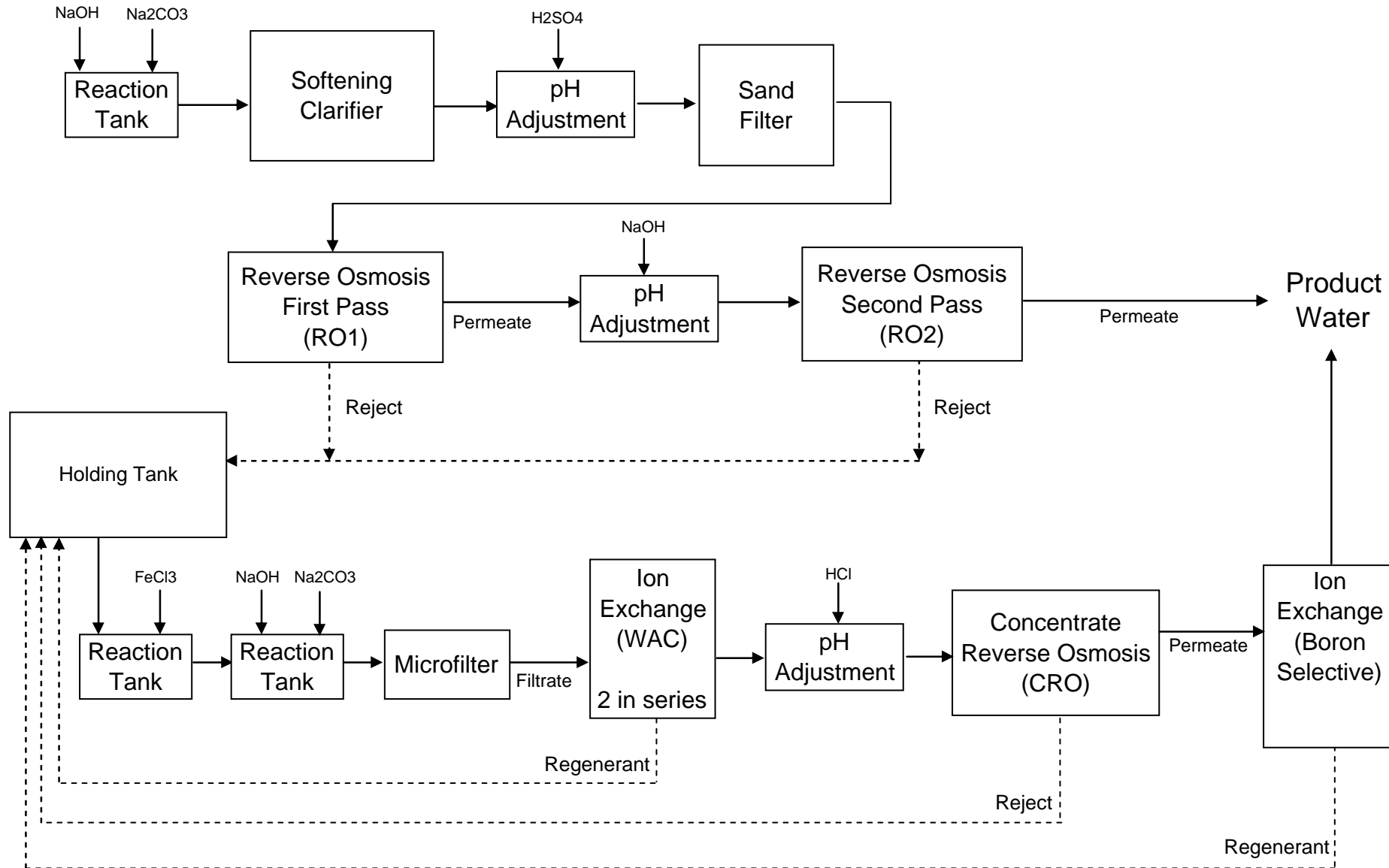
The discharge from the primary RO subsystem will be combined with the discharge from the boron specific ion exchange column to form the discharge from the system. Regenerants from the ion exchange columns and reject from the concentrate RO subsystem will be recycled back to the beginning of that subsystem. Blowdown from the system is removed in two places; in the microfilter as precipitated hardness and heavy metal hydroxides and in the reject from the concentrate RO (approximately 88% is permeate and 12% is rejected).

A block flow diagram of the overall system is presented in Figure 1.

In addition to meeting the very tight discharge requirements of numerous parameters, other challenges of this study were the removal of compounds such as boron and ammonia, both of which can not be precipitated and require specific pH's for removal with reverse osmosis.

The overall system goal is purification of at least 97% of the water. The remaining 3% would require evaporation.

Figure 1 - Block Diagram of Treatment System



SECTION II EXECUTIVE SUMMARY

Based on the laboratory testing conducted as part of this study, the proposed treatment system should allow for compliance with the discharge requirements provided to Siemens by Foth & Van Dyke with the possible exception of mercury..

- The permeate from the first pass of the primary RO system met all performance criteria with the exception of boron. This was expected. Boron was subsequently removed in the second pass primary RO. Most of the parameters were below the performance criteria by at least one order of magnitude.
- The permeate from the concentrate RO (CRO) was also of very good quality, however, there were three parameters for which the performance criteria were exceeded. Comments on each are provided below:

- Boron - It was anticipated that the boron levels in the CRO permeate would exceed the discharge criteria, therefore, the CRO permeate was polished using a boron selective resin. As long as the CRO was operated at a pH of 10.5, the effluent from the boron specific resin was well within the performance criteria.
- Ammonia - When the CRO is operated at pH 10.5, ammonia levels in the permeate exceeded the performance criteria. However, when this stream is blended with the primary RO permeate, the overall discharge criterion is met. If an additional safety factor is needed, testing showed that ammonia can be completely removed from the system by incorporating breakpoint chlorination as part of the pretreatment prior to microfiltration.
- Mercury - Due to a technician's error, mercury results were only obtained for the first of the three stages that were conducted for the CRO. The feed sample for stages 2 and 3 was prepared incorrectly and seriously compromised all of the mercury related data for these two stages. More importantly, repeated attempts to clean the system were not effective in removal of residual mercury contamination.

The mercury level in the RO permeate obtained in stage 1 CRO was 2.5 ppt, which exceeded the performance criteria of 2.0 ppt. As with ammonia, once it was blended with the permeate from the primary RO, the overall criteria for mercury were met. However, it should be kept in mind that due to contamination, testing was not able to demonstrate that the mercury limit could be met in stage 2 and 3 of the CRO, which would include the recycle streams and would be more representative of normal operation. Testing is currently underway to evaluate the use of a mercury selective resin to further polish the CRO permeate prior to blending with the primary RO stream.

SECTION III SAMPLE DESCRIPTION

Due to the fact that the mine does not yet exist, all samples used in these tests were laboratory prepared. The composition of the laboratory prepared sample was based on a projected analysis provided by Foth Infrastructure and Environment.

All samples were generated in a 500-gallon fiberglass tank using laboratory grade chemicals and deionized water. Table 1 provides a listing of the concentrations of various constituents that are expected to be found in the wastewater. Table 2 is a listing of the compounds used for preparing the synthetic sample as well as an analysis of the actual laboratory prepared sample.



Client: Kennecott Minerals Company Scope ID.: 04W018
 Project: Eagle Project
 Prepared by: JFF1 Date: 05/16/06
 Checked by: _____ Date: _____

Benchscale Test Treatment Paramaters (Draft)

	WWTP Influent Wastewater (µg/L)	Part 22 Std (µg/L)	Influent % of Part 22 (%)	Include in bench scale test?
Antimony	19	3	626%	Yes
Arsenic	33	25	131%	Yes
Barium	28	1,000	3%	No
Beryllium	1.0	2	50%	No
Boron	3,671	250	1468%	Yes
Cadmium	11	2.5	448%	Yes
Calcium	63,345	n.a.		Yes
Chloride	825,963	250,000	330%	Yes
Chromium	8.5	50	17%	No
Cobalt	652	20	3258%	Yes
Copper	145	500	29%	Yes, (for surface water limit issues)
Fluoride	706	1,000	71%	No
Iron	6,467	300	2156%	Yes
Lead	9.0	2	448%	Yes
Lithium	85	85	100%	No
Magnesium	32,317	200,000	16%	Yes
Manganese	992	50	1984%	Yes
Mercury	0.0410			Yes, (for surface water limit issues)
Molybdenum	21	18.5	112%	Yes
Nickel	33,403	50	66805%	Yes
Nitrogen, Ammonia	10,163	5,000	203%	Yes
Nitrogen, Nitrate	50	5,000	1%	No
Phosphorus, total	18.5	1,000	2%	No
Potassium	9,842	n.a.		Yes
Selenium	26	25	102%	No
Silver	4.3	17	25%	No
Sodium	411,536	120,000	343%	Yes
Strontium	2,031	2,300	88%	No
Sulfate	167,099	250,000	67%	Yes
Thallium	7.1	1	714%	Yes
Vanadium	6.3	2	288%	Yes

Table 2 - Sample Preparation and Initial Analysis

Ion	Compound Used	Target (as ion) (ug/l)	Actual Analysis (ug/l)	Difference (ug/l)	Difference (%)
Ammonia-N	NH ₄ Cl	10,163	11,000	837	7.6%
Antimony	SbF ₃	19	14.2	-4.8	-33.8%
Arsenic	Na ₂ HAsO ₄ ·7H ₂ O	33	37	4	10.8%
Barium	BaCl ₂ ·2H ₂ O	28	28.2	0.2	0.7%
Beryllium	Be(NO ₃) ₂ ⁽¹⁾	1	0.9	-0.1	-11.1%
Boron	H ₃ BO ₃	3,671	3,610	-61	-1.7%
Cadmium	CdSO ₄	11	8.4	-2.6	-31.0%
Calcium	CaCl ₂ ·2H ₂ O	63,345	74,900	11,555	15.4%
Chloride	various salts ⁽²⁾	825,963	878,000	52,037	5.9%
Cobalt	CoCl ₂ ·6H ₂ O	652	585	-67	-11.5%
Copper	CuCl ₂ ·2H ₂ O	145	130	-15	-11.5%
Iron	FeCl ₃ ·6H ₂ O	6,467	6,320	-147	-2.3%
Lead	PbCl ₂	9	14	5	36.2%
Magnesium	MgCl ₂ ·6H ₂ O	32,317	30,400	-1,917	-6.3%
Manganese	MnCl ₂ ·4H ₂ O	992	764	-228	-29.8%
Mercury	HgCl ₂	0.041	0.081	0.04	49.4%
Molybdenum	Na ₂ MoO ₄ ·2H ₂ O	21	14	-7	-50.0%
Nickel	NiCl ₂ ·6H ₂ O	33,403	33,600	197	0.6%
Potassium	KCl	9,842	29,900	20,058	67.1%
Selenium	Na ₂ SeO ₄	26	30.8	4.8	15.6%
Silver	AgNO ₃	4.3	4.2	-0.1	-2.4%
Sodium	various salts ⁽²⁾	411,536	424,000	12,464	2.9%
Strontium	SrCl ₂ ·6H ₂ O	2,031	2,980	949	31.8%
Sulfate	Na ₂ SO ₄	167,099	177,000	9,901	5.6%
Thallium	TlF ₃	7.1	6	-1.1	-18.3%
Vanadium	Na ₃ VO ₄	6.3	7.5	1.2	16.0%
Zinc	ZnCl ₂	351	304	-47	-15.5%

Notes:

(1) A 10,000 ppm Be in 5% AA standard was used.

(2) Both chloride and sodium were added through the addition of various counter ions.

SECTION IV PRIMARY REVERSE OSMOSIS SUBSYSTEM

A. Process Description

Wastewater generated at the mine will be pretreated using softening chemistry, sodium carbonate and sodium hydroxide, and then settled in a clarifier. The overflow from the clarifier will be passed through a sand filter for removal of residual suspended solids. The sand filter effluent will then be passed through two passes of reverse osmosis. Permeate from the primary RO will be directed to the secondary RO. The permeate from the second RO will be discharged. Rejects from both RO's will be combined and treated in the concentrate treatment system (see Section V).

The first step in the study will be to conduct a series of jar tests to optimize the removal of hardness and heavy metals. Once the chemistry has been optimized, the treatment will be repeated on a larger scale, generating a sufficient volume of softened water to allow for processing through a laboratory scale RO.

B. Pretreatment / Softening

1. Jar Tests

Softening involves the addition of sodium carbonate at an elevated pH for removal of calcium and magnesium. Calcium precipitates out as calcium carbonate and magnesium is removed in the form of magnesium hydroxide. Various heavy metals including iron and nickel will also be removed as insoluble hydroxides.

A series of jar tests were conducted to determine the optimum chemical doses. Softening calculations were conducted to determine the dosages for the initial jar tests. Based on the results of the initial tests, additional tests were conducted to minimize chemical consumption and optimize removal. The target goal was to reduce the calcium to less than 14 mg/l and the magnesium to less than 8 mg/l.

The following general procedure was used for all jar tests:

- Add sodium carbonate
- Add either lime or sodium hydroxide
- Mix for 10 minutes
- Filter through glass fiber filter paper (1.5 μ)

Results

All chemical doses and analytical results are presented in Table 3.

Based upon the results achieved in jar testing, the treatment scheme that most closely matched the target results was 450 mg/l of sodium carbonate and sodium hydroxide to pH 11.

Table 3 – Jar Testing Results

Treatment	Sodium Carbonate Added (mg/l)	Sodium Hydroxide Added (mg/l)	Lime Added (mg/l)	TSS ⁽¹⁾ (mg/l)	Effluent Analysis			
					Final pH	Calcium (mg/l)	Magnesium (mg/l)	TSS (mg/l)
None	---	---	---	---	---	73	34	6
Lime	325	---	347	500	11.2	101	< 0.1	11.24
Lime	500	---	350	---	11.1	8.3	0.97	11.12
Lime	450	---	230	580	11	29.4	2.3	10.99
Sodium Hydroxide	325	250	---	208	11.2	47	0.52	11.19
Sodium Hydroxide	350	125	---	---	10.8	48	18.7	10.78
Sodium Hydroxide	400	123	---	---	10.8	37.3	18.5	10.82
Sodium Hydroxide	450	200	---	150	11	11.3	5	10.98
Sodium Hydroxide	500	175	---	---	11.2	8.6	0.98	11.15
(1) - TSS analysis conducted on treated sample prior to settling (to estimate sludge generation)								

2. Process Simulation

In order to simulate the softening process which would take place in a reactor clarifier, it is desirable to treat several small batches of sample. Following treatment of each batch, a heel of sludge is purposely left in the reactor. This heel helps to “seed” the precipitation of calcium carbonate during the processing of subsequent batches.

a. Equipment Description

Tank MaterialPolyethylene
Tank Dimensions36” diameter x 48” deep
MixerElectric, 1750 rpm, with 30” stainless steel shaft

b. Treatment

To increase the effectiveness of the softening process, 500 gallons of simulated sample was treated in three separate batches

Batch 1

163 gallons of simulated wastewater was added to the reaction tank. Sodium carbonate was added first (450 mg/l). Sodium hydroxide was then added to pH 11 (50% liquid NaOH used). The solution was mixed for thirty minutes and an anionic polymer was added to enhance settling (2 mg/l Alumafloc I). The solution was mixed for 3 minutes and then settled for 60 minutes.

The supernatant was then pumped into the sand filter feed tank. Analysis showed it contained 15 mg/l of total suspended solids.

Batch 2

155 gallons of untreated feed was added to the softening tank containing 20 gallons of sludge generated during treatment of batch 1. This mixture was treated and decanted in the same manner as batch 1.

Batch 3

170 gallons of untreated feed was added to the softening tank containing 25 gallons of sludge generated during treatment of batch 2. This mixture was treated and decanted in the same manner as batches 1 and 2.

Prior to decanting, the solution was mixed for thirty minutes and analyzed for total suspended solids (816 mg/l). An anionic polymer was added and the

solution was mixed for 3 minutes and then settled for 60 minutes. This supernatant contained 14 mg/l of total suspended solids.

C. Sand Filtration

1. Equipment Description

Column Material.....Clear PVC
Column Size.....3" diameter x 60" deep
Bed Depth (sand).....36"
Base (quartz).....4"
Service Type.....Downflow
Service Flow Rate.....12 gpm/ft²
Feed Pump.....Peristaltic
Connections.....Tygon Tubing

2. General Operation

Prior to using the sand filter it was thoroughly backwashed with deionized water (until no fines were seen in the backwash water).

Sand filtration was conducted on the supernatant from the softening test described above (Section IV.B.2). The supernatant following settling was pumped downflow through the column. During the first 50 minutes of operation, a steady increase of pressure was observed (up to 26 psi). It is believed that the pressure increase was due to the precipitation of calcium carbonate (post-precipitation) occurring due to the high surface area of the sand particles and elevated pH. To prevent this from occurring, the pH of the sand filter feed was lowered to 7.8 with sulfuric acid.

Prior to restarting, the sand column was back-washed and the top 2" of sand was removed from the column. Upon restarting, the pressure quickly increased again. The column was backwashed again and another 2" of sand was removed. This time the column ran for 5.5 hours before the pressure increased again (only to 13.5 psi this time). The column was backwashed once again. No sand was removed this time. This time the sand filter ran for 7 hours with very little pressure increase noted. It is believed that time was needed to flush all of the calcium carbonate from the system.

Operating data obtained from the running of the sand filter is presented in Table 4. A complete analysis of the sand filter effluent is presented in Table 7 (located near the end of Section D, below).

Table 4 - Sand Filter Operating Data

Time (Minutes)	Flow Rate (lpm)	GPM/ft2	Gallons Processed	Pressure (psi)	Notes
0	2	10.8		4	Run 1 - pH 11
10	2	10.8	5.3	10	
20	2	10.8	5.3	14	
30	2	10.8	5.3	19	
40	2	10.8	5.3	24	
50	2	10.8	5.3	26	Stop and backwash - remove top 2" of media
0	2	10.8		5.5	Run 2 - pH adjust to 7.8 with H ₂ SO ₄ (~ 185 mg/l H ₂ SO ₄ required)
10	2	10.8	5.3	8	
20	2	10.8	5.3	16	
30	2	10.8	5.3	20	
40	2	10.8	5.3	26	Stop and backwash - remove top 2" of media
0	2	10.8		1	Run 3 - pH adjust to 7.8 with H ₂ SO ₄ (~ 185 mg/l H ₂ SO ₄ required)
60	2	10.8	31.7	1.5	
120	2	10.8	31.7	2.5	
180	2	10.8	31.7	4.5	
240	2	10.8	31.7	5.5	
330	2	10.8	47.6	13.5	Stop and backwash
0	2	10.8		1	
45	2	10.8	23.8	1.5	
420	2	10.8	198.2	2.5	Run 4 - pH adjust to 7.8 with H ₂ SO ₄ (~ 185 mg/l H ₂ SO ₄ required)
Total gallons processed:			444		

D. Reverse Osmosis

The effluent from the sand filter was collected and composited as a single sample. It was then processed through a two-pass RO system. The same RO was used for both passes. Permeate from the first pass was collected in a holding tank, and then processed through a second time to simulate the second pass. To flush the equipment between passes, the permeate and reject obtained from processing the first 50 gallons through the second pass were discarded.

1. Equipment Description

RO elements	Three, 2.5 in x 40 inch each
Element type	Brackish water, thin-film composite
Surface area	28 ft ² per element
Pump type	Positive displacement



Figure 2 - Photograph of the Reverse Osmosis Unit

2. General Operation

A sketch showing the operation of the RO used for these tests is shown in Figure 3

a. First Pass (RO1)

The entire sand filter effluent was composited into a single tank and the pH was adjusted to 6.5 with hydrochloric acid. This RO was operated at a flux rate of 10 gallon/day/ft² (FGD) and a permeate recovery rate of approximately 75%.

b. Second Pass (RO2)

The permeate from the first pass RO (RO1) was collected as a single sample and then pH adjusted to 10.5 with sodium hydroxide. The higher pH is required for the effective removal of boron. This RO was also operated at a flux rate of 10 gallon/day/ft² (FGD) however the permeate recovery rate was targeted at 85%.

3. Results

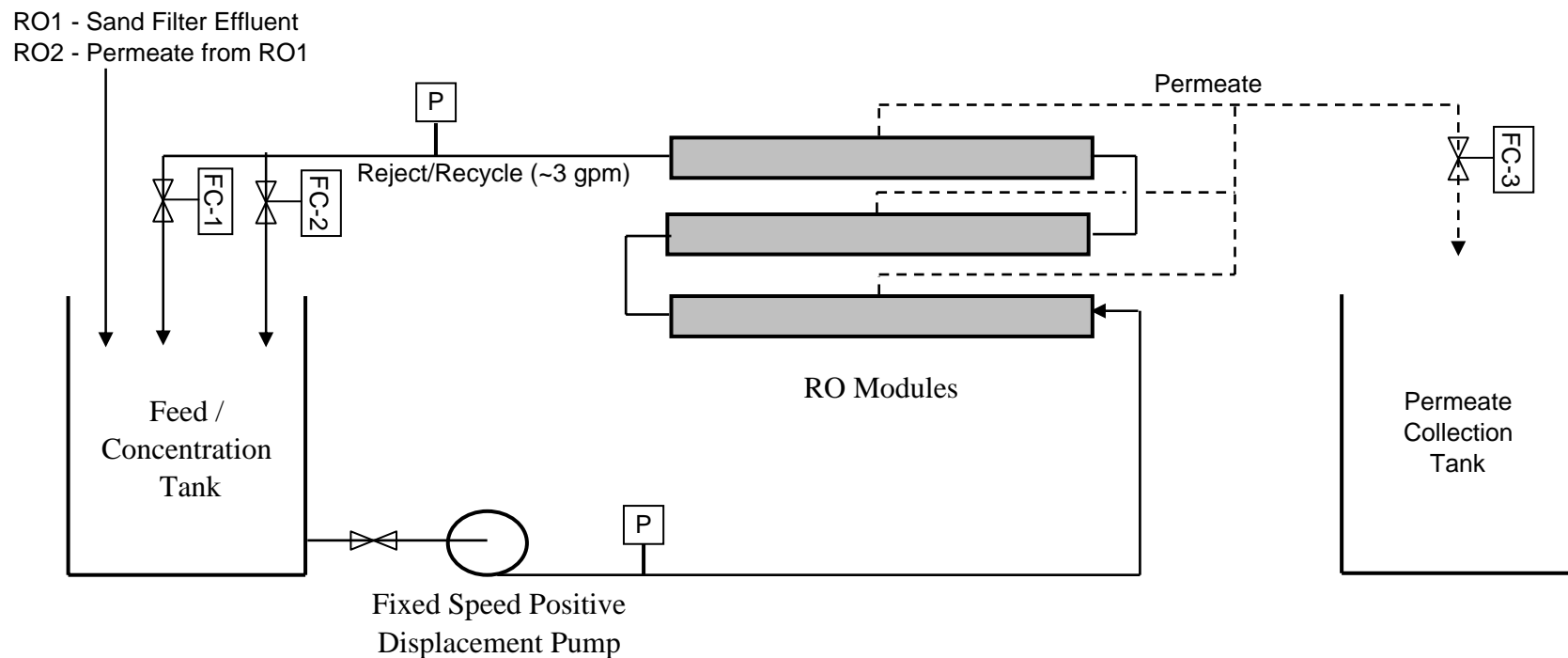
a. Operating Data

Table 5 and 6

b. Analytical Data

The permeates from both the first pass and second pass RO runs as well as the combined rejects were analyzed for selected parameters. The results can be found in Table 7.

Figure 3 - Operation of RO1 and RO2



1. The "feed/concentration" tank is filled with a known amount of sample.
2. Flow control valves FC-1 and FC-3 are closed and FC-2 is full open.
3. Flow is initiated to the positive displacement pump.
4. Valves FC-1 and FC-3 are gradually opened and FC-2 is gradually closed until the desired permeate and reject flow rates are obtained.
5. Permeate is collected in the collection tank. Reject is returned to the "feed/concentration" tank.
6. The system is operated until the desired concentration factor is achieved.

For RO1 - the permeate rate was 2200 ml/min and the reject rate was 730 ml/min

For RO2 - the permeate rate was 2300 ml/min and the reject rate was 410 ml/min

Table 5 - First Pass RO (RO1) Operating Data

Operating Time (minutes)	Influent Pressure (psi)	Effluent Pressure (psi)	ΔP (psi)	Permeate Flow (mL/min)	Reject Flow (mL/min)	Total Gallons Processed	Actual Flow (gfd)	Actual Recovery (percent)	Temp. (C)
0	155	110	45	2,210	730	0	10.0	75.2	
30	155	110	45	2,210	700	23	10.0	75.9	20.5
60	155	110	45	2,210	730	46	10.0	75.2	20.6
120	175	130	45	2,250	710	93	10.2	76.0	20.8
180	175	130	45	2,280	710	141	10.3	76.3	20.9
240	180	140	40	2,300	710	188	10.4	76.4	
300	180	140	40	2,280	720	236	10.3	76.0	20.6
360	180	140	40	2,200	730	282	10.0	75.1	20.6
420	200	160	40	2,200	720	329	10.0	75.3	20.6
480	240	200	40	2,250	720	376	10.2	75.8	21.2
500	300	260	40	2,230	720	391	10.1	75.6	21.4
<div> <div> <u>Target Operating Conditions</u> Flux rate = 10 gfd Permeate flow = 2208 ml/min Reject Flow = 736 ml/min </div> <div> <u>Operating Data</u> Total gallons processed = 391 Feed Conductivity (mmhos/cm) = 3,740 Permeate Conductivity (mmhos/cm) = 131 Reject Conductivity (mmhos/cm) = 13,600 </div> </div>									

Table 6 - Second Pass RO (RO2) Operating Data

Operating Time (minutes)	Influent Pressure (psi)	Effluent Pressure (psi)	ΔP (psi)	Permeate Flow (mL/min)	Reject Flow (mL/min)	Total Gallons Processed	Actual Flow (gfd)	Actual Recovery (percent)	Temp. (C)
0	140	110	30	2,300	410	0	10.4	84.9	
60	145	115	30	2,300	400	43	10.4	85.2	20.6
120	150	120	30	2,300	410	86	10.4	84.9	20.7
180	150	120	30	2,300	410	129	10.4	84.9	20.8
240	150	120	30	2,300	410	172	10.4	84.9	21.1
300	150	120	30	2,280	405	214	10.3	84.9	21.5
<div> <div> <u>Target Operating Conditions</u> Flux rate = 10 gfd Permeate flow = 2300 ml/min Reject Flow = 410 ml/min </div> <div> <u>Operating Data</u> Total gallons processed = 214 Feed Conductivity (mmhos/cm) = 131 Permeate Conductivity (mmhos/cm) = 52 Reject Conductivity (mmhos/cm) = 816 </div> </div>									

Table 7 - Primary RO Sub-system Analytical Data

	Feed	Softened	Sand	RO1	RO2	Combined
Ion	Analysis	Water	Filtrate	Permeate	Permeate	Reject
	(ug/l)	(ug/l)	(ug/l)	(ug/l)	(ug/l)	(ug/l)
Ammonia - N	11,000	na	6,220	549	578	17,700
Antimony	14.2	na	27.4	<0.60	< 0.60	0.62
Arsenic	37	na	3.03	<0.60	< 0.60	4.61
Barium	28.2	na	2.30	< 2.0	< 2.0	14.8
Beryllium	0.9	na	< 0.10	< 0.10	< 0.10	< 0.10
Boron	3,610	na	3,960	2,280	62.4	5,850
Cadmium	8.4	na	< 0.20	< 0.20	< 0.20	0.35
Calcium	74,900	10,600	1,600	330	0.15	13,900
Chloride	878,000	na	838,000	30,700	1,930	1,390,000
Cobalt	585	na	2.73	< 0.60	< 0.60	19.0
Copper	130	na	1.51	0.74/<0.6	3.14	4.30
Fluoride	706	na	< 100	< 100	< 100	< 100
Iron	6,320	na	25.0	<10	< 10	< 10
Lead	14.1	na	<0.30	<0.30	0.84	0.45
Magnesium	30,400	1,500	1,300	< 100	< 100	8,420
Manganese	764	na	4.0	1.30	< 1.0	27.10
Mercury	81 ppt	11 ppt	3.6 ppt	< 0.13 ppt	0.64 ppt	7.3 ppt
Molybdenum	14	na	42.8	< 0.60	< 0.60	9.10
Nickel	33,600	na	153	3.10	0.50	1,350
Potassium	29,900	na	28,900	2,710	< 1,000	49,600
Selenium	30.8	na	33.2	< 0.60	< 0.60	20.0
Silver	4.2	na	<0.2	< 0.20	< 0.20	< 6
Sodium	424,000	na	859,000	21,600	4,460	1,310,000
Strontium	2,980	na	447	1.25	< 0.60	1,610
Sulfate	177,000	na	171,000	< 1,000	< 1,000	883,000
Thallium	6	na	11.6	< 0.20	< 0.20	10.0
Vanadium	7.5	na	< 1.0	< 1.0	< 1.0	< 1.0
Zinc	304	na	3.07	6.16	8.01	31.5

E. Discussion

- Other than the initial plugging problems related to calcium carbonate build-up in the sand filter, testing of the primary RO subsystem was very successful. To prevent plugging in the sand filter it must be operated at a pH of less than 8.
- All discharge requirements, with the exception of boron were easily met following the first pass RO.
- Raising the pH to 10.5 prior to second pass RO allowed for compliance with the discharge requirement for boron.
- There was a very slight increase in the copper, lead, mercury, and zinc levels during second pass RO. The cause for this increase is unknown and is believed to be the results of either an analytical error or due to laboratory contamination.

SECTION V CONCENTRATE REVERSE OSMOSIS SUBSYSTEM

A. Overview

The feed stream used for this portion of testing was the combined reject from RO1 and RO2. This subsystem consisted of the following unit processes:

- Pretreatment (softening chemistry)
- Microfiltration
- Ion exchange (weak acid cation)
- pH adjustment
- Reverse osmosis (referred to as the concentrate RO or CRO, as it will be treating reject from the primary RO's; RO1 and RO2.)
- Boron selective ion exchange resin

In the full scale system, the wastewater obtained from the regeneration of both ion exchange columns as well as the reject from the concentrate RO will all be combined with the feed stream to this subsystem. In simulating this in the lab, there are no rejects or regenerants until the system has been operating for several days. Therefore, testing was conducted in three separate stages.

Also, it is important to note that in order to minimize the quantity of water that needed to be processed through RO1 and RO2, all testing of the concentrate RO subsystem was done on simulated RO reject. The simulated water was prepared based on the actual analysis of combined RO1 and RO2 rejects (presented in Table 7 - previous section)

The CRO testing was conducted in three stages:

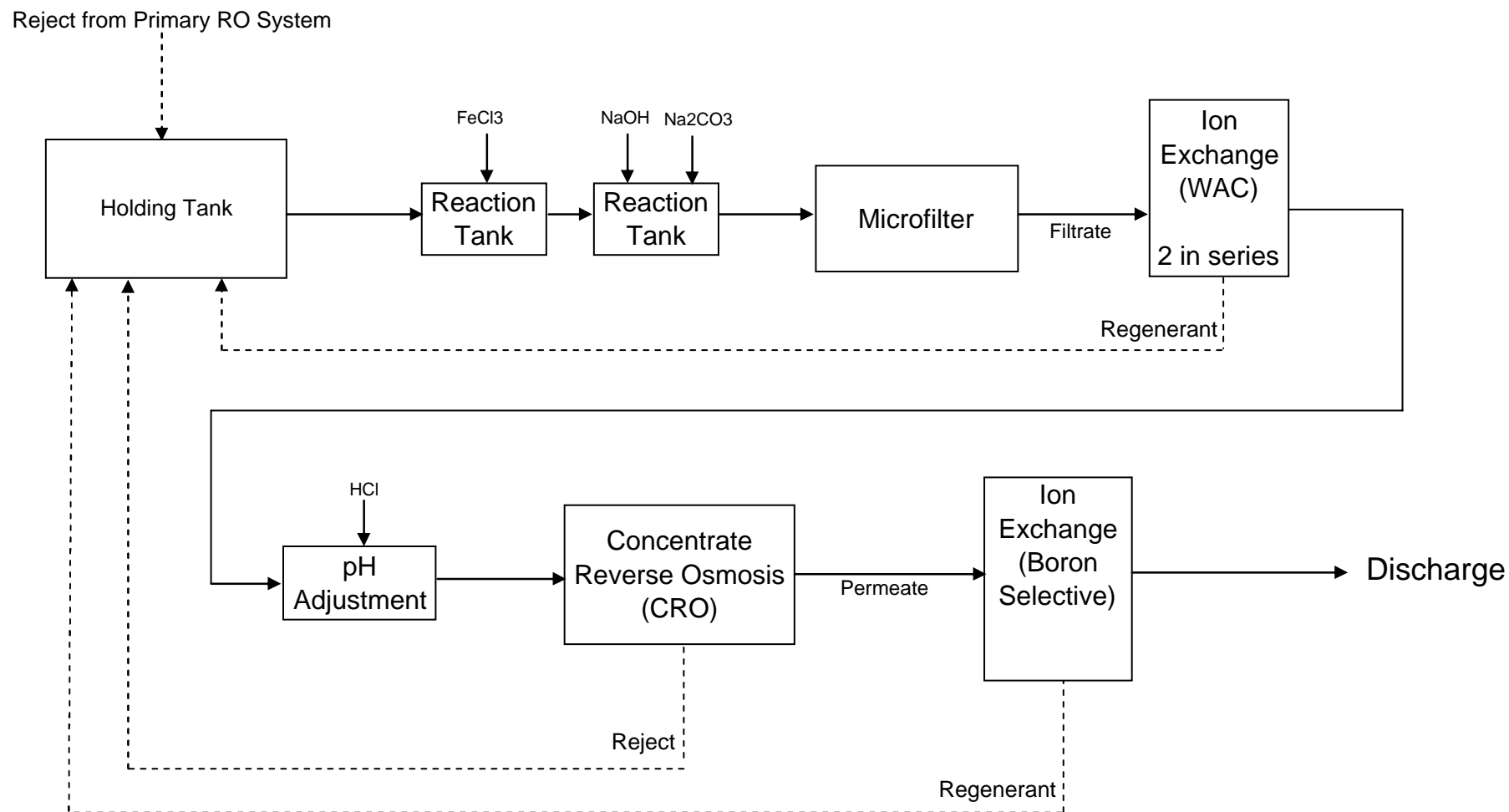
Stage 1 - During this stage of testing, the feed consisted of the simulated combined RO1 and RO2 reject only. No recycled wastestreams were used. This stage was conducted until the weak acid cation (WAC) column was fully loaded and required regeneration.

Stage 2 - Stage 2 was operated in the same manner as stage 1, however, the ion exchange regenerants and the reject from the "concentrate" RO which were generated in stage 1 were added to the feed (ahead of the microfilter).

Stage 3 - Stage 3 was operated in the same manner as stage 2, with the ion exchange regenerants and the reject from the "concentrate" RO which were generated in stage 2 added to the feed (ahead of the microfilter).

A sketch of this subsection is presented in Figure 4.

Figure 4 - Block Diagram of Concentrate RO (CRO) System



B. Sample Preparation

The main constituent used in all three stages described above is the combined reject from RO1 and RO2. In order to obtain a sufficient volume of this solution (1500 gallons), nearly 15,000 gallons of water would need to be processed through RO1 and RO2. The laboratory is not equipped to easily handle volumes of this magnitude; therefore laboratory prepared RO1/RO2 reject was used for all CRO testing. Table 8 provides a listing of the chemicals and quantities used for preparing this solution.

1. Stage 1

As mentioned above, the stage 1 sample contained simulated primary RO reject only (Table 9). No recycled wastestreams were used.

2. Stage 2

This sample was prepared as follows:

- 340 gallons of simulated primary RO reject (Table 8)
- 160 gallons of concentrate RO (CRO) reject
- 9 gallons of WAC regenerant (see Section V.C.2.c)
- 0.69 gallons of simulated boron IX regenerant (see Section V.C.3.c)

Mercury Contamination

In the process of preparing Stage 2 feed, a technician error was made. Instead of preparing the solution to contain 10 ng/l of mercury, it was accidentally prepared to contain 480,000 ng/l. This unfortunate error was not realized until analytical results were received approximately 2 weeks after the error was made. As a result, all mercury related data for Stage 2 and Stage 3 is not applicable and must be discarded. It is our belief that this error affected only the values for mercury. No other data appeared to be compromised.

Upon learning of the error, at the completion of Stage 3, the entire treatment system was completely drained and thoroughly cleaned with acid and detergents. The cleaning included replacement of the RO and microfilter membranes, replacing interconnecting tubing, and changing out of the resin in both the WAC and boron ion exchange columns. This was done three separate times. Unfortunately, the level of mercury found during each subsequent re-start of the system was in excess of an acceptable level to resume testing.

3. Stage 3

This sample was prepared as follows:

- 340 gallons of simulated primary RO reject (Table 8)
- 160 gallons of concentrate RO (CRO) reject
- 9 gallons of WAC regenerant (see Section V.C.2.c)
- 0.69 gallons of simulated boron IX regenerant (see Section V.C.3.c)

Table 8 - Preparation of CRO Feed Solution

		Target	Stock Solution		Stage 1			Stages 2 & 3	
		Concentration	as Ion		Quantity			Quantity	
Ion	Chemical used	(ug/l)	(g/l)		Added ⁽¹⁾	Units		Added ⁽²⁾	Units
Ammonia-N	NH ₄ Cl	17,700.00	337.2		99	g		67.3	g
Antimony	SbF ₃	0.62	1		1.2	mls		0.8	mls
Arsenic	Na ₂ HAsO ₄ ·7H ₂ O	4.61	10		0.9	mls		0.6	mls
Barium	BaCl ₂ ·2H ₂ O	14.80	1		28	mls		19.0	mls
Beryllium	Be(NO ₃) ₂	0.10	10		0.02	mls		0.0	mls
Boron	H ₃ BO ₃	5,850.00	174.8		63	g		42.8	g
Cadmium	CdSO ₄	0.35	1		0.7	mls		0.5	mls
Calcium	CaCl ₂ ·2H ₂ O	13,900.00	272.6		96	g		65.3	g
Chloride	various salts ⁽³⁾	1,390,000.00	---		---	---		---	---
Cobalt	CoCl ₂ ·6H ₂ O	19.00	10		3.6	mls		2.4	mls
Copper	CuCl ₂ ·2H ₂ O	4.30	10		0.8	mls		0.5	mls
Iron	FeCl ₃ 6 H ₂ O	10.00	20.6		0.9	g		0.6	g
Lead	PbCl ₂	0.45	1		0.9	mls		0.6	mls
Magnesium	MgCl ₂ ·6H ₂ O	8,420.00	119.6		133	g		90.4	g
Manganese	MnCl ₂ ·4H ₂ O	27.10	100		0.5	mls		0.3	mls
Mercury	HgCl ₂	0.01	0.01		1.4	mls		0.95	mls
Molybdenum	Na ₂ MoO ₄ ·2H ₂ O	9.10	1		17	mls		11.6	mls
Nickel	NiCl ₂ ·6H ₂ O	1,350.00	247		10.3	g		7.0	g
Potassium	KCl	49,600.00	524.4		179	g		121.7	g
Selenium	Na ₂ SeO ₄	20.00	0.418		90.6	mls		61.6	mls
Silver	AgNO ₃	6.00	0.635		17.9	mls		12.2	mls
Sodium	various salts ⁽³⁾	1,310,000.00	393.3		6,304	g		4286.7	g
Strontium	SrCl ₂ ·6H ₂ O	1,610.00	100		30.5	mls		20.7	mls
Sulfate	Na ₂ SO ₄	883,000.00	676.3		2,471	g		1680.3	g
Thallium	TlF	10.00	0.915		21	mls		14.3	mls
Vanadium	Na ₃ VO ₄	1.00	1		1.9	mls		1.3	mls
Zinc	ZnCl ₂	31.50	4.797		12.4	mls		84.5	mls

(1) Added to a total volume of 500 gallons

(2) Added to a total volume of 340 gallons

(3) Both chloride and sodium were added through the addition of various counter ions.

C. Process Description

1. **Microfiltration**

The first step in the treatment process is softening / microfiltration. It is in this step that hardness (calcium and magnesium) and other heavy metals are removed from the system as precipitated sludge. This step is very important due to the fact that it prevents the buildup of hardness caused by the recycling of ion exchange regenerants and RO reject.

a. Equipment Description

The following is a general description of the microfilter used for testing. (See Figure 5 for a photograph of the microfilter, Figure 6 for a block flow diagram, and Figure 7 for an overhead photograph of the microfilter and reaction tanks).

Membrane Material of construction	Polymeric
Quantity	2
Surface area, total	3.0 sq. ft.
Pore size (nominal microns)	0.2
Concentration tank operating volume.....	5-20 gallons
Pump type	Centrifugal

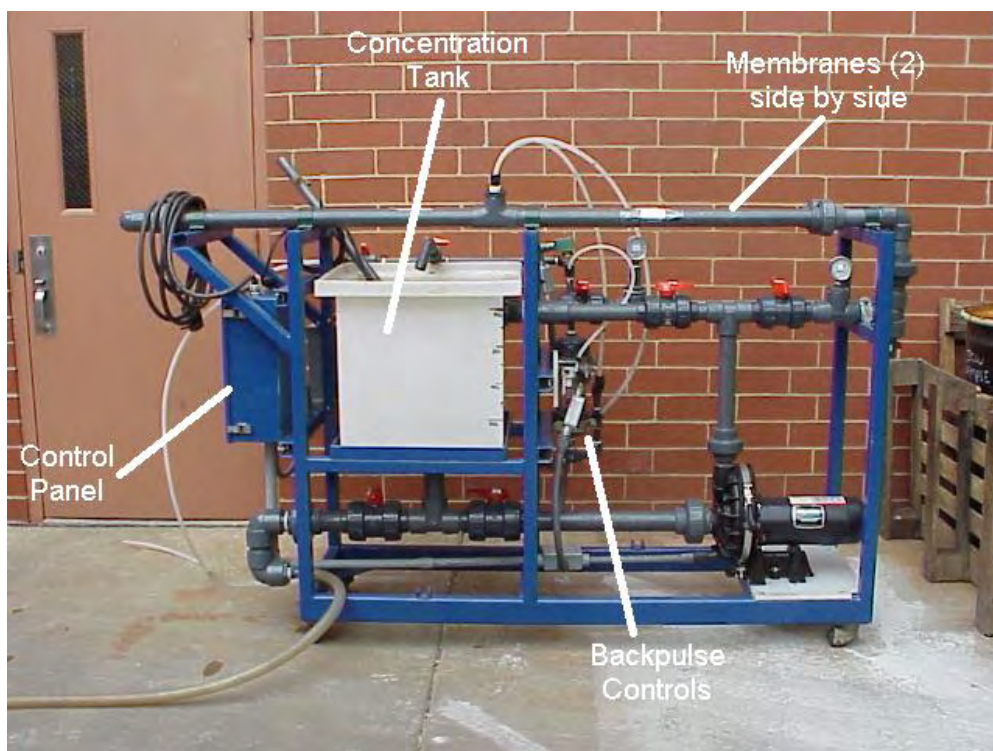


Figure 5 - Photograph of the Microfilter (without reaction tanks).

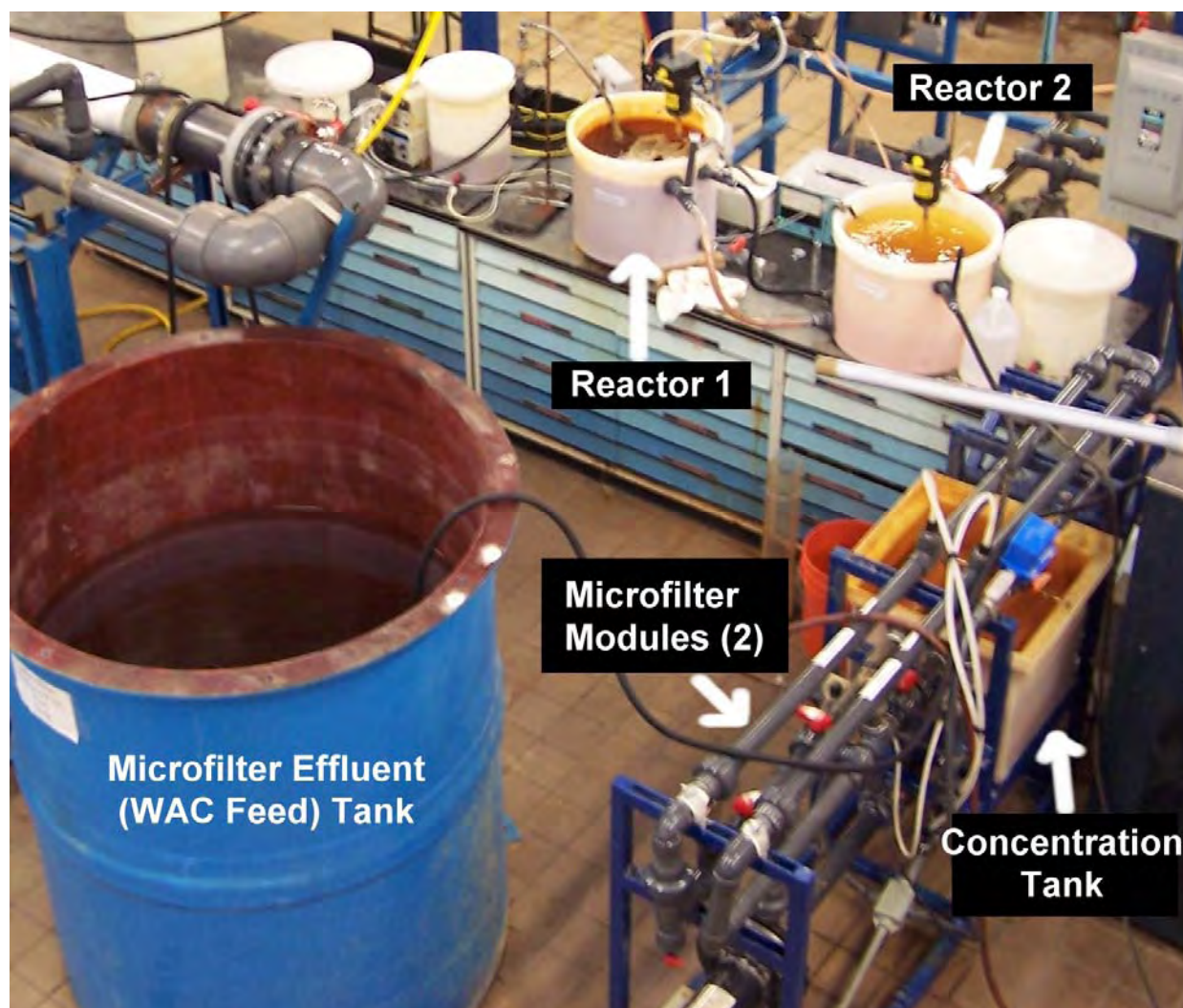
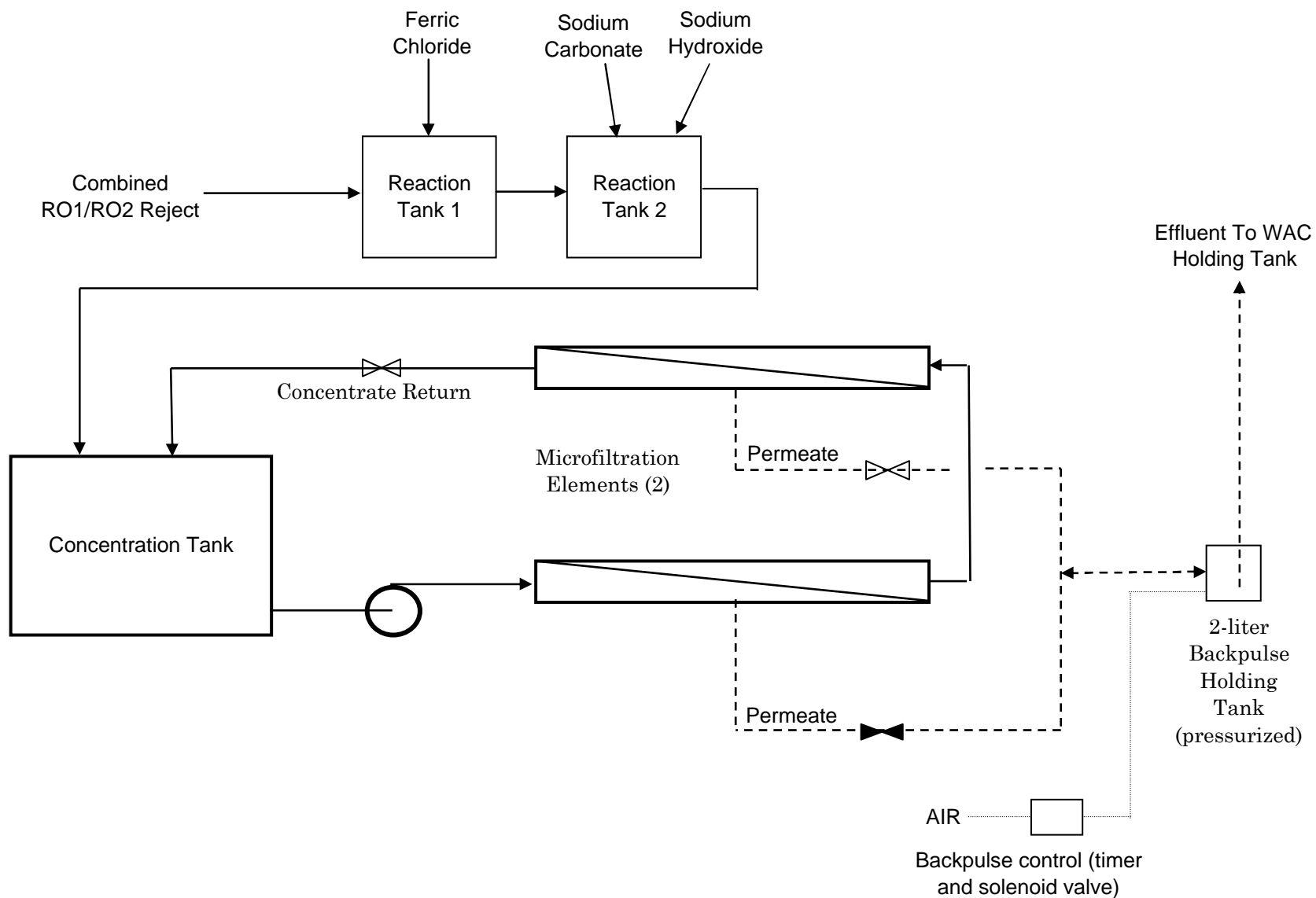


Figure 6 - Photograph of Microfilter with Reaction Tanks

Figure 7
Block Diagram of Microfilter System



b. Process Description

1. Jar Tests

In order to determine the optimum chemistry for operation of the microfilter, a series of jar tests were conducted using various doses of sodium carbonate and sodium hydroxide. The following general procedure was used for all jar tests:

- Add sodium carbonate
- Add sodium hydroxide
- Mix for 15 minutes
- Filter through glass fiber filter paper (1.5 μ)

The results are presented in Table 9.

Table 9 - Microfilter Jar Tests

Test	Na ₂ CO ₃ (mg/l)	Target pH	TSS ⁽¹⁾ mg/l	Filtrate Analysis		
				Calcium (mg/l)	Magnesium (mg/l)	Actual pH
Non e	na	na	na	60	29	5.2
1	250	10.8	na	47	16	10.82
2	300	10.8	na	25	17	10.78
3	400	10.8	130	13.6	16.5	10.82
4	400	11	135	13.2	3.9	10.98
5	400	11.2	136	9	0.6	11.25
(1) - TSS was from a treated sample prior to settling						

2. Treatment Chemistry

The following pretreatment chemistry was selected based on jar testing results and was used in the continuous flow system.

Reactor 1

- Add 15 mg/l ferric chloride
- Add hydrochloric acid to pH = 4.5
- 15 minute retention time

Reactor 2

- Add 400 mg/l sodium carbonate
(sodium carbonate was only needed when the feed contained WAC
regenerant - none was added at other times.)
- Add sodium hydroxide to pH 11
- 15 minute retention time

The discharge from Reactor 2 flows into the concentration tank of the microfilter from where it is pumped through the microfilter modules.

3. Operating Data

See Table 10 for operating data relating to the microfilter system. In viewing the data it should be noted that the feed to the microfilter was a continuous flow of 2.5 liters per minute. The microfilter cycled on and off since water processed through the filter at an average rate of 878 gfd (7.1 liters per minute).

c. Results

Analysis showing the feed and the discharge from the microfiltration system for each of the three stages is presented in Table 11. Also, at the end of this section, a complete analysis across all unit processes is presented (Tables 20-22).

Table 10 - Microfilter Operating Data

Run Time ⁽¹⁾ (hours)	Inlet Pressure (psi)	Outlet Pressure (psi)	Flux (gfd)	Concentrate TSS (mg/l)	Temp (C)	Comments
0	28	20	2,054	---	19.6	Clean water flux
4	28	20	951	7,000	20.5	
10	28	20	989	---	20.6	
28	28	20	989	---	20.6	
34	28	20	837	---	20.4	
40	28	20	837	13,280	20.6	End Stage 1
44	28	20	837	---	20.1	
48	28	20	862	---	19.8	
68	28	20	818	---	---	
72	28	20	761	---	---	
86	28	20	812	---	---	
90	28	20	850	24,120	---	End Stage 2
88	28	20	913	---	20.5	
100	28	20	888	---	---	
102	28	20	913	---	---	
116	28	20	888	---	---	
118	28	20	888	---	---	
120	28	20	888	33,180	---	End Stage 3
<p>(1) Run Time - the values presented are total run time and include time that the microfilter idled while waiting for additional feed to enter the concentration tank. The feed to the microfilter was 2.5 liters per minute, flux rates averaged 7.1 liters per minute, indicating that the microfilter ran about 35% of the time.</p>						

Table 11 - Microfilter Feed and Effluent

Cycle	1		3	
	Feed	Effluent	Feed	Effluent
Antimony	0.62	<3	<3	<3
Arsenic	4.61	<3	4.22	<3
Barium	14.8	12.9	26.8	<10
Beryllium	< 0.10	<0.5	<0.5	<0.5
Boron	5,850	5,770	17,100	15,900
Cadmium	0.35	<1	<1	<1
Calcium	13,900	16,800	38,400	14,900
Cobalt	19.0	<3	8.74	<3
Copper	4.30	<3	5.96	3.68
Iron	< 10	58.0	87.0	<10
Lead	0.45	<1.5	<1.5	2.06
Magnesium	8,420	2,370	14,900	120
Manganese	27.1	<5	9.90	<5
Mercury	7.3 ppt	2.0 ppt	na	na
Molybdenum	9.10	9.52	12.80	12.10
Nickel	1,350	95.9	653	6.16
Potassium	49,600	71,600	78,000	72,700
Selenium	20.0	17.8	24.2	24
Silver	< 6	<1	11.5	1.38
Sodium	1,310,000	1,740,000	2,890,000	3,010,000
Strontium	1,610	1,740	4,940	3,490
Thallium	10.0	9.63	11.9	10.8
Vanadium	< 1.0	<5	<5	<5
Zinc	31.5	16.3	na	na
Ammonia -N	17,700	11,400	11,000	10,500
Chloride	1,390,000	1,960,000	3,490,000	3,530,000
Fluoride	< 100	< 100	<100	<100
Sulfate	883,000	927,000	1,080,000	1,040,000
na - not analyzed due to incorrect preparation of feed				

2. Weak Acid Cation Ion Exchange

The effluent from the microfilter was collected in a 470 gallon FRP tank. From there it was pumped through a weak acid cation (WAC) exchange column for removal of residual heavy metals and hardness prior to reverse osmosis.

a. Equipment Description

Columns.....	2 in series
Column material	Clear PVC
Column Size	2" diameter x 72" deep
Resin	Lanxess CNP 80 WS
Resin Bed Depth	36" (hydrogen form)
Resin Volume	1850 ml (0.065 ft ³)
Resin Form	Hydrogen
Service Flow	Downflow
Service Flow Rate.....	2.4 gpm/ft ³
Feed Pump.....	Peristaltic
Connections	Tygon tubing

b. General Operation

Microfilter filtrate was pumped through the weak acid cation ion exchange columns (WAC) at 2.4 gpm/ft³. This was continued until the calcium concentration in the discharge from the primary column exceeded 1 mg/l. When this happened, the system was stopped and the column was regenerated.

Each of the three stages was considered complete when the WAC column began to leak calcium at a level greater than 1 mg/l.

It should be noted that during the loading cycle, the resin swelled from 36 inches to 54 inches.

c. Regeneration

Regeneration was accomplished using hydrochloric acid. Prior to regeneration, the column was backwashed with deionized water for 10 minutes using a flow rate of 1 gpm/ft³. The backwash did not contain any noticeable solids and was directed back to the WAC feed tank.

The regeneration and subsequent rinses were all countercurrent (upflow). All regeneration solutions, including rinses were saved and recycled back to the feed tank prior to the microfilter. A portion of the first two regeneration solutions was also analyzed. The results are presented in Table 14.

Regenerant.....	20 g/l HCl
Regenerant Volume	17.8 liters (9.6 BV)
Regenerant Flow Rate	1 gpm/ft ³
Slow Rinse Volume	3.6 liters (1.94 BV)
Slow Rinse Flow Rate	3 gpm/ft ²
Fast Rinse Volume	13.25 liters (7.14 BV)
Fast Rinse Flow rate.....	6 gpm/ft ²

d. Results

Samples were collected at regular intervals to monitor breakthrough of calcium and magnesium. These results are shown in Table 12.

A complete analysis of the feed and the discharge from the weak acid cation system for each of the three stages is presented in Table 13. Also, at the end of this section, a complete analysis across all unit processes is presented (Tables 20-22).

Table 12 - Calcium and Magnesium Leakage from WAC Column

Volume Processed (gallons)	Influent			Column 1 Effluent				Column 2 Effluent				Column 1 Loading Data			
	Ca (mg/l)	Mg (mg/l)		Ca (mg/l)	Mg (mg/l)	pH		Ca (mg/l)	Mg (mg/l)	pH		Ca Loaded (grams)	Ca Leaked (grams)	Mg Loaded (grams)	Mg Leaked (grams)
Stage 1															
10	14	8		< 0.1	< 0.1	2.7		< 0.1	< 0.1	2.8		0.5	0.0	0.3	0.0
25	14	8		< 0.1	< 0.1	2.81		< 0.1	< 0.1	2.99		1.3	0.0	0.8	0.0
50	14	8		< 0.1	< 0.1	3.38		< 0.1	< 0.1	3.12		2.6	0.0	1.5	0.0
75	14	8		< 0.1	< 0.1	3.39		< 0.1	< 0.1	3.14		4.0	0.0	2.3	0.0
125	14	8		< 0.1	< 0.1	3.56		< 0.1	< 0.1	3.4		6.6	0.0	3.8	0.0
150	14	8		< 0.1	< 0.1	3.65		< 0.1	< 0.1	3.45		7.9	0.0	4.5	0.0
200	14	8		< 0.1	< 0.1	3.77		< 0.1	< 0.1	3.54		10.6	0.0	6.1	0.0
315	14	8		< 0.1	< 0.1	4.02		< 0.1	< 0.1	3.71		16.7	0.0	9.5	0.0
390	14	8		0.018	< 0.1	6.16		< 0.1	< 0.1	3.86		20.7	0.0	11.8	0.0
420	14	8		< 0.1	< 0.1	6.69		< 0.1	< 0.1	3.84		22.3	0.0	12.7	0.0
455	14	8		< 0.1	< 0.1	7.87		< 0.1	< 0.1	3.85		24.1	0.0	13.8	0.0
500	14	8		< 0.1	< 0.1	9.7		< 0.1	< 0.1	4.05		26.5	0.0	15.1	0.0
680	19	9		< 0.1	< 0.1	10.1		< 0.1	< 0.1	3.9		39.4	0.0	21.3	0.0
860	19	9		< 0.1	0.1	10.8		< 0.1	< 0.1	4.4		52.4	0.0	27.4	0.1
1,000	19	9		< 0.1	0.1	11.1		< 0.1	< 0.1	4.6		62.5	0.0	32.2	0.1
1,180	19	9		< 0.1	0.2	11.2		< 0.1	< 0.1	6.6		75.4	0.0	38.3	0.1
1,350	19	9		0.1	0.4	11.3		< 0.1	< 0.1	7.1		87.6	0.1	44.1	0.3
1,450	19	9		0.15	0.6	11.3		< 0.1	< 0.1	10.2		94.8	0.1	47.5	0.2
1,500	19	9		1.4	0.75	11.3		< 0.1	< 0.1	10.2		98.4	0.3	49.2	0.1
Stage 1 Totals												98.4	0.5	49.2	0.8

Table 12 - Calcium and Magnesium Leakage from WAC Column

Volume Processed (gallons)	Influent		Column 1 Effluent			Column 2 Effluent			Column 1 Loading Data			
	Ca (mg/l)	Mg (mg/l)	Ca (mg/l)	Mg (mg/l)	pH	Ca (mg/l)	Mg (mg/l)	pH	Ca Loaded (grams)	Ca Leaked (grams)	Mg Loaded (grams)	Mg Leaked (grams)
Stage 2												
300	7	8	< 0.1	< 0.1	6.2	< 0.1	< 0.1	9.8	7.9	0.0	9.1	0.0
500	19	8	< 0.1	< 0.1	10.1	< 0.1	< 0.1	9.9	14.4	0.0	15.1	0.0
750	21	9	< 0.1	< 0.1	11	< 0.1	< 0.1	10.9	34.3	0.0	23.7	0.0
1,000	21	9	0.1	0.1	11.2	< 0.1	< 0.1	11.1	54.1	0.1	32.2	0.1
1,250	19	8	0.2	0.2	11.2	< 0.1	< 0.1	11.3	72.1	0.0	39.7	0.2
1,350	19	8	0.7	0.3	11.3	< 0.1	< 0.1	11.2	79.3	0.3	42.8	0.1
1,400	19	8	1.2	0.3	11.2	< 0.1	< 0.1	11.2	75.7	0.2	44.3	0.1
Stage 2 Totals									75.7	0.5	44.3	0.4
Stage 3												
300	5	5	< 0.1	< 0.1	6.3	< 0.1	< 0.1	9.6	5.7	0.0	5.7	0.0
500	19	8	< 0.1	< 0.1	9.5	< 0.1	< 0.1	10.1	14.4	0.0	15.1	0.0
750	20	9	< 0.1	< 0.1	10.7	< 0.1	< 0.1	11.2	33.3	0.0	23.7	0.0
1,000	20	9	0.1	0.1	11.2	< 0.1	< 0.1	11.2	52.2	0.1	32.2	0.1
1,250	19	9	0.1	0.2	11.2	< 0.1	< 0.1	11.1	70.2	0.0	40.7	0.2
1,350	19	9	0.5	0.3	11.1	< 0.1	< 0.1	11.2	77.4	0.1	44.1	0.1
1,400	19	9	1.6	0.5	11.2	< 0.1	< 0.1	11.3	73.8	0.3	45.8	0.1
Stage 3 Totals									73.8	0.4	45.8	0.4
SUMMARY												
			Stage 1			Stage 2			Stage 3			
			Loaded (lbs/ft³)	Removed (lbs/ft³)	Recovery (per cent)	Loaded (lbs/ft3)	Removed (lbs/ft³)	Recovery (per cent)	Loaded (lbs/ft3)	Removed (lbs/ft³)	Recovery (per cent)	
Calcium			3.30	3.22	97.70	2.53	2.40	94.70	2.47	No regeneration		
Magnesium			1.63	1.23	75.60	1.48	1.25	84.50	1.53	No regeneration		

Table 13 - WAC Feed and Effluent

Cycle	1		2		3	
	<u>Feed</u>	<u>Effluent</u>	<u>Feed</u>	<u>Effluent</u>	<u>Feed</u>	<u>Effluent</u>
Antimony	<3	3.46	na	1.08	<3	<3
Arsenic	<3	<3	na	1.39	<3	3
Barium	12.9	<10	na	2.0	<10	<10
Beryllium	<0.5	<0.5	na	<0.1	<0.5	<0.5
Boron	5,770	5,690	na	14,300	15,900	16,500
Cadmium	<1	<1	na	<0.2	<1	<1
Calcium	16,800	340	na	<100	14,900	190
Cobalt	<3	<3	na	<0.6	<3	<3
Copper	<3	<3	na	2.53	3.68	73.6
Iron	58.0	<10	na	<10	<10	<10
Lead	<1.5	<1.5	na	<0.3	2.06	<1.5
Magnesium	2,370	<100	na	<100	120	<100
Manganese	<5	<5	na	<1	<5	<5
Mercury	2.0 ppt	2.9 ppt	na	14,000 ppt	na	na
Molybdenum	9.52	9.21	na	10.70	12.10	11.1
Nickel	95.9	4.24	na	3.01	6.16	2.86
Potassium	71,600	70,800	na	56,600	72,700	67,500
Selenium	17.8	17.5	na	18.5	24	22.8
Silver	<1	<1	na	3.240	1.38	<1
Sodium	1,740,000	1,700,000	na	2,400,000	3,010,000	2,860,000
Strontium	1,740	7.84	na	0.66	3,490	<3
Thallium	9.63	6.18	na	3.49	10.8	<1
Vanadium	<5	<5	na	<1	<5	<5
Zinc	16.3	<10	na	na		na
Ammonia -N	11,400	na	na	na	10,500	na
Chloride	1,960,000	na	na	na	3,530,000	na
Fluoride	< 100	na	na	na	<100	na
Sulfate	927,000	na	na	na	1,040,000	na
na - not analyzed (cycle 2 feed sample was accidentally destroyed prior to analysis).						

Table 14 - Analysis of WAC Regenerant Solutions

	Units	Stage 1	Stage 2
Antimony	µg/l	37	< 12.0
Arsenic	µg/l	96.9	< 12.0
Barium	µg/l	2,060	800
Beryllium	µg/l	< 5	< 2.00
Boron	µg/l	618	351
Cadmium	µg/l	< 10	< 4.00
Calcium	mg/l	2,850	1,230
Cobalt	µg/l	501	12.7
Copper	µg/l	207	82.7
Iron	mg/l	8.08	1.00
Lead	µg/l	102	< 6.00
Magnesium	mg/l	747	514
Manganese	µg/l	734	20
Molybdenum	µg/l	< 30	< 12.0
Nickel	µg/l	29	0.82
Potassium	mg/l	50.9	45.1
Selenium	µg/l	< 30	< 12.0
Silver	µg/l	220	335
Sodium	mg/l	1,150	2,690
Strontium	mg/l	251	163
Thallium	µg/l	106	113
Vanadium	µg/l	< 50	< 20

3. Concentrate Reverse Osmosis (CRO)

a. Equipment Description

The same RO that was used for simulation of the primary RO treatment was used here (see Section IV.D.1).

b. General Operation

See Figure 8 for a diagram and information on operation of the concentrate RO (CRO). The discharge from the secondary WAC column was collected until approximately 100 gallons was accumulated. This volume was then pH adjusted to 10.5 using either sodium hydroxide or hydrochloric acid.

The dosage of neutralizing agent varied significantly based on the pH of the WAC column effluent, which also varied significantly. The WAC effluent was initially acidic as sodium and calcium ions replaced the hydrogen ions which were present following regeneration. The pH gradually increased as the hydrogen ions were depleted from the resin and it began to release sodium ions. The pH varied from a low of 2.8 to a high of 11.3 during the three stages. Additional pH information regarding the pH of the WAC effluent can be found on Table 12.

Operating data as well as quantity of neutralizing chemical required is presented in Table 15.

Figure 8 - Operation of CRO

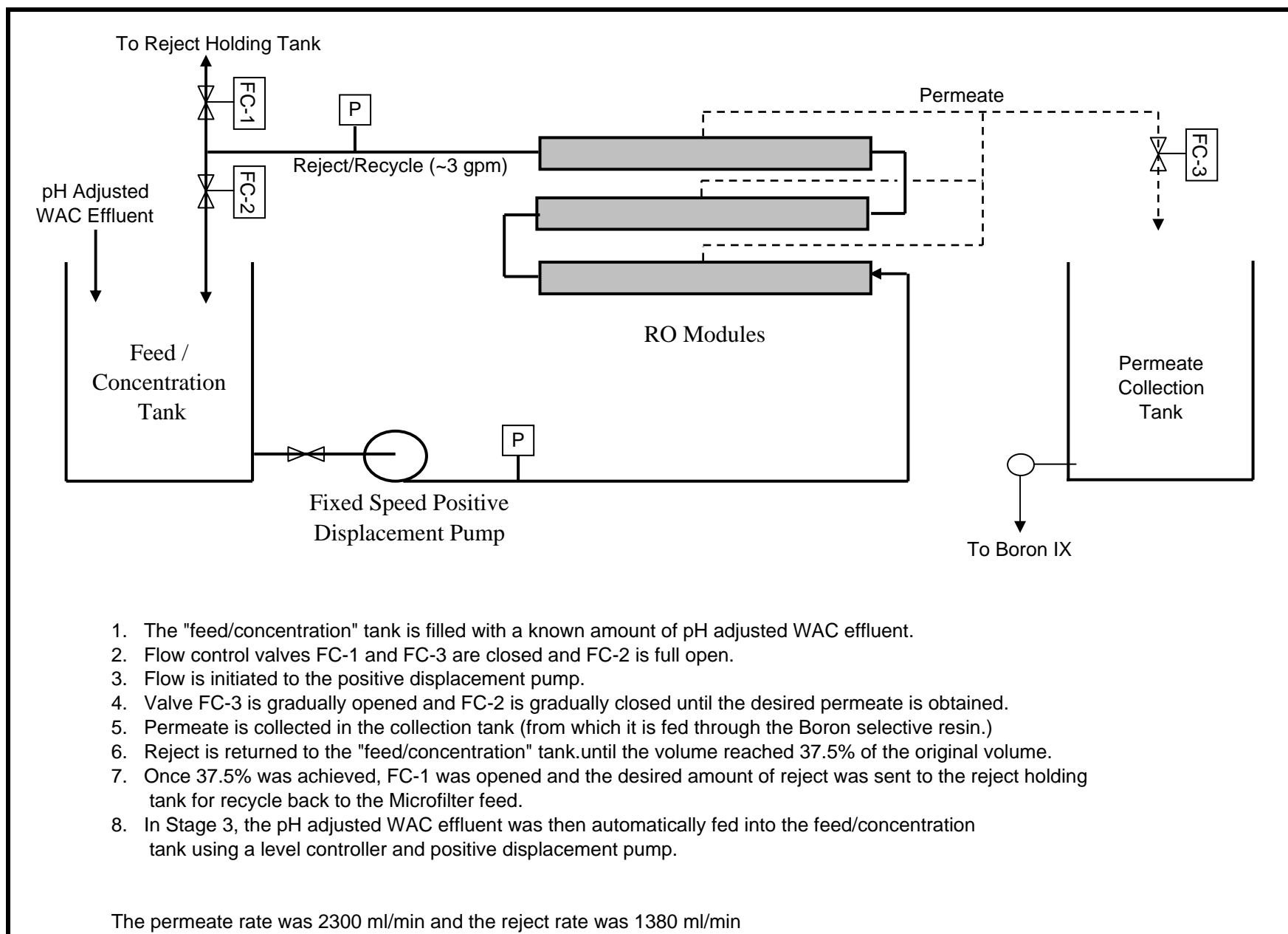


Table 15 - CRO Operating Data

Operating Time (Hours)	Influent Pressure (psi)	Effluent Pressure (psi)	Differential (psi)	Permeate Flow (mL/min)	Reject Flow (mL/min)	Actual Flow (gfd)	Actual Recovery (percent)	Temp. (C)	Total Gallons Processed	NaOH (mg/l)	HCl (mg/l)
0	220	200	20	2,210	730	10.0	75.2	20.2	0	---	---
---	---	---	---	---	---	---	---	---	100	234	---
4	230	210	20	2,210	700	10.0	75.9	20.5	185	nr	nr
---	---	---	---	---	---	---	---	---	200	201	---
---	---	---	---	---	---	---	---	---	300	184	---
---	---	---	---	---	---	---	---	---	400	182	---
---	---	---	---	---	---	---	---	---	500	60	---
12	240	220	20	2,210	730	10.0	75.2	20.6	557	nr	nr
---	---	---	---	---	---	---	---	---	600	---	5
---	---	---	---	---	---	---	---	---	700	---	46
---	---	---	---	---	---	---	---	---	800	---	52
---	---	---	---	---	---	---	---	---	900	---	64
32	230	210	20	2,250	710	10.2	76.0	20.8	1,496	nr	nr
BEGIN STAGE 2											
0	280	270	10	2,210	730	10.0	75.2	20.2	0	nr	nr
4	290	280	10	2,210	700	10.0	75.9	20.5	185	nr	nr
---	---	---	---	---	---	---	---	---	500	---	51
12	310	290	20	2,210	730	10.0	75.2	20.6	557	nr	nr
---	---	---	---	---	---	---	---	---	600	---	64
---	---	---	---	---	---	---	---	---	700	---	81
30	260	250	10	2,250	710	10.2	76.0	20.8	1,402	nr	nr
BEGIN STAGE 3											
0	330	310	20	2,200	730	10.0	75.1	20.2	0	nr	nr
4	330	310	20	2,200	740	10.0	74.8	20.5	186	nr	nr
8	330	310	20	2,220	735	10.1	75.1	20.6	374	nr	nr
12	330	310	20	2,240	730	10.1	75.4	20.6	562	nr	nr
16	330	310	20	2,240	730	10.1	75.4	20.6	750	nr	nr
28	340	320	20	2,250	710	10.2	76.0	20.8	1,314	nr	nr
30	340	320	20	2,250	710	10.2	76.0	20.8	1,407	nr	nr

c. Ammonia/Boron Removal

Stages 1 and 2 tests were conducted as described in the text above. Stage 3 was started in a similar manner to the first two stages; pH adjusted to 10.5 with caustic and normal operation. However, during stage 3 analytical data was received which showed that ammonia exceeded the anticipated discharge requirements.

Upon receipt of that analysis, stage 3 testing was interrupted to evaluate the effect that adjusting the pH of the feed to the CRO had on both ammonia and boron. In order to minimize the volume of solution affected by these tests the contents of the feed/concentration tank were withdrawn to the minimum volume necessary to safely operate the unit (~25 gallons). During testing, the permeate and reject flows were directed back into the feed/concentration tank to minimize the volume of water required for testing.

The RO was operated at three separate pH's. The permeate at each pH was analyzed for ammonia and boron. Results are presented in Table 16

Table 16 - Effect of pH on CRO Permeate

pH	Ammonia-N (mg/l)	Boron (mg/l)
10.5	10.6	0.3
9.5	7	3.5
8.5	1.3	4.5

Upon completion of this pH evaluation, normal testing was resumed, however, at pH 9.5 instead of 10.5. Operation was maintained in this manner until approximately 150 gallons had been processed. At this point it was decided that breakpoint chlorination would be used prior to the microfiltration stage and operation was resumed at pH 10.5.

d. Breakpoint Chlorination

As mentioned above, breakpoint chlorination was evaluated for removal of ammonia from the system. Several jar tests were conducted to determine the effectiveness of using it as part of the microfiltration pretreatment. The following tests were done on a sample of simulated primary RO reject, with CRO reject and ion exchange regenerants added:

- Adjust pH to 7.5 with sodium hydroxide
- Add sodium hypochlorite
- Stir for 15 minutes
- Analyze for ammonia

Table 17 - Breakpoint Chlorination

NaOCl added (<u>mg/l</u>)	Residual NH ₄ -N (<u>mg/l</u>)
0	8.0
80	7.5
120	<0.2
240	<0.2

e. Results

Analysis showing the feed and the discharge from the concentrate RO system for each of the three stages is presented in Table 18. Also, at the end of this section, a complete analysis across all unit processes is presented (Tables 21-23).

It should be noted that the effluent samples whose results are presented in Tables 18 and 23 were collected during the period that the CRO was operated at a reduced pH, therefore the boron numbers are higher than they would be if the pH was raised to 10.5 as planned.

Table 18 - CRO Feed and Effluent

Cycle	1		2		3		Draft Monthly Average *	Report Only
	Feed	Effluent	Feed	Effluent	Feed	Effluent	(ug/l)	(ug/l)
Antimony	3.46	<0.6	1.08	<0.6	<3	<0.6	---	5
Arsenic	<3	<0.6	1.39	<0.6	3	<0.6	6	---
Barium	<10	<2	2.0	<2	<10	<2	---	7
Beryllium	<0.5	<0.1	<0.1	<0.1	<0.5	<0.1	---	0.25
Boron	5,690	253	14,300	624	16,500	7,120	250	---
Cadmium	<1	<0.2	<0.2	<0.2	<1	<0.2	3	---
Calcium	340	250	<100	670	190	210	---	---
Cobalt	<3	<0.6	<0.6	<0.6	<3	<0.6	---	46
Copper	<3	<0.6	2.53	<0.6	73.6	1.61	10	---
Iron	<10	<10	<10	18.0	<10	<10	---	16
Lead	<1.5	0.51	<0.3	<0.3	<1.5	<0.3	---	2.5
Magnesium	<100	<100	<100	<100	<100	<100	---	---
Manganese	<5	2.60	<1	<1	<5	<1	---	12
Mercury	2.9 ng/l	2.5 ng/l	14,000 ng/l	2,600 ng/l	na	na	2.1 ng/l	---
Molybdenum	9.21	<0.6	10.70	<0.6	11.1	<0.6	---	5.5
Nickel	4.24	<0.3	3.01	4.18	2.86	<0.3	---	24.5
Potassium	70,800	2,590	56,600	2,520	67,500	2,280	---	6,000
Selenium	17.5	<0.6	18.5	<0.6	22.8	<0.6	5	---
Silver	<1	<0.2	3.240	<0.2	<1	<0.2	0.4	---
Sodium	1,700,000	58,000	2,400,000	75,100	2,860,000	72,300	---	150,000
Strontium	7.84	<0.6	0.66	21.2	<3	<0.6	---	4.75
Thallium	6.18	<0.2	3.49	<0.2	<1	<0.2	---	2
Vanadium	<5	<1	<1	<1	<5	<1	---	1.5
Zinc	<10	14.4	na	14.4	na	na	---	85
Ammonia -N	na	10,600	na	6,850	na	5,420	5,000	---
Chloride	na	69,600	na	52,400	na	52,400	---	220,000
Fluoride	na	< 100	na	<100	na	<100	---	205
Sulfate	na	2,980	na	2,040	na	2,040	---	8,500

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Draft Permit No.GW1810162

na Not analyzed

4. Boron Selective Ion Exchange

The permeate from the CRO was collected in a 200 gallon polypropylene tank. From there it was pumped through a boron selective ion exchange column for removal of residual boron prior to “discharge”.

a. Equipment Description

Columns.....	1
Column material	Clear PVC
Column Size	3” diameter x 72” deep
Resin	Lanxess MK-51
Resin Bed Depth	40” (hydroxide / chloride form)
Resin Volume	4630 ml (0.163 ft ³)
Resin Form	hydroxide / chloride
Service Flow	Downflow
Service Flow Rate.....	2 gpm/ft ³
Feed Pump.....	Peristaltic
Connections	Tygon tubing

b. General Operation

CRO permeate was collected in a 200 gallon holding tank, then pumped downflow through the boron selective ion exchange column at 2 gpm/ft³. No pH adjustment was done between the CRO and the boron resin.

Water was to be processed until the boron concentration in the effluent exceeded 1 mg/l. However, this never occurred until Stage 3, and that was only while the pH of the feed was lowered to 9.5. As a result, regeneration of this column was not necessary.

c. Regeneration

Although regeneration of the boron column was not required during our tests, it will be required at some point in the full scale system. Based on loading information from the manufacturer and on the quantity of boron in CRO permeate, it was estimated that regeneration would be required approximately every 30 days.

A synthetic regenerant was prepared based on the assumption that the resin would be loaded to 50% of its theoretical capacity at the time regeneration

would be required (186 g is theoretical loading at full capacity). The following is the chemicals and volumes of rinse water that would be typically used for regeneration.

Regenerant 1	80 g/l HCl
Regenerant 1 - Volume	7.5 liters
Rinse 1 - Volume	6.8 liters
Regenerant 2	30 g/l NaOH
Regenerant 2 - Volume	9.9 liters
Rinse 2 - Volume	27.9 liters

Based on the above information, the synthetic boron ion exchange regenerant was produced as follows:

7.5 liters of 80 g/l hydrochloric acid
9.9 liters of 30 g/l sodium hydroxide
34.7 liters of deionized water
532 g of boric acid (H_3BO_3)

It was assumed that the regenerant and rinses would be placed in a storage tank and bled into the CRO system over a 20 day period.

d. Results

Samples were collected at regular intervals to monitor breakthrough of boron. These results are shown in Table 19.

A complete analysis of the feed and the discharge from the weak acid cation system for each of the three stages is presented in Table 20. Also, at the end of this section, a complete analysis across all unit processes is presented (Tables 21-23).

Table 19 - Boron Leakage from Boron Selective Resin Column

Volume Processed (gallons)	Influent		Effluent		Loading Data	
	Boron (mg/l)	pH			Boron Loaded (grams)	Boron Leaked (grams)
Stage 1						
5	0.27	10.49	< 0.2		0.0	0.0
15	0.27	10.49	< 0.2		0.0	0.0
30	0.27	10.49	< 0.2		0.0	0.0
50	0.27	10.49	< 0.2		0.1	0.0
250	0.27	10.52	<0.02		0.3	0.0
300	0.27	10.52	<0.02		0.3	0.0
400	0.2	10.53	0.023		0.3	0.0
600	0.2	10.53	<0.02		0.5	0.0
700	0.26	10.59	<0.02		0.7	0.0
900	0.26	10.54	< 0.02		0.9	0.0
Stage 1 Totals					3.0	0.0
Stage 2						
200	0.6	10.64	< 0.02		0.5	0.0
300	0.6	10.62	< 0.02		0.7	0.0
450	0.55	10.48	< 0.02		0.9	0.0
600	0.55	10.59	0.029		1.2	0.0
750	0.48	10.58	0.025		1.4	0.0
850	0.48	10.56	< 0.02		1.5	0.0
Stage 2 Totals					6.2	0.0
Stage 3						
150	7.1	9.44	0.9		5.6	0.5
300	7.1	9.5	1.2		13.6	1.2
Stage 3 Totals					19.2	1.7

Table 20 - Boron Ion Exchange Influent and Effluent

Cycle	1		2		3		Draft Monthly Average *	Report Only
	Feed	Effluent	Feed	Effluent	Feed	Effluent	(ug/l)	(ug/l)
Antimony	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	---	5
Arsenic	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	6	---
Barium	<2	<2	<2	<2	<2	<2	---	7
Beryllium	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	---	0.25
Boron	253	<6	624	<6	7,120	1,160	250	---
Cadmium	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	3	---
Calcium	250	280	670	510	210	280	---	---
Cobalt	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	---	46
Copper	<0.6	<0.6	<0.6	<0.6	1.61	<0.6	10	---
Iron	<10	<10	18.0	<10	<10	10.0	---	16
Lead	0.51	<0.3	<0.3	<0.3	<0.3	0.32	---	2.5
Magnesium	<100	<100	<100	<100	<100	<100	---	---
Manganese	2.60	<1	<1	<1	<1	<1	---	12
Mercury	2.5 ppt	na	2,600 ppt	820 ppt	na	na	2.1 ng/l	---
Molybdenum	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	---	5.5
Nickel	<0.3	6.18	4.18	0.55	<0.3	1.52	---	24.5
Potassium	2,590	3,390	2,520	1,950	2,280	2,100	---	6,000
Selenium	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	5	---
Silver	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	0.4	---
Sodium	58,000	77,400	75,100	87,900	72,300	69,300	---	150,000
Strontium	<0.6	1.71	21.2	16.7	<0.6	1.18	---	4.75
Thallium	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	---	2
Vanadium	<1	1.6	<1	<1	<1	<1	---	1.5
Zinc	14.4	na	14.4	14.4	na	na	---	85
Ammonia -N	10,600	9,930	6,850	7,570	5,420	4,870	5,000	---
Chloride	69,600	136,000	52,400	97,800	52,400	97,800	---	220,000
Fluoride	< 100	<100	<100	<100	<100	<100	---	205
Sulfate	2,980	1,750	2,040	4,140	2,040	3,180	---	8,500

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na Not analyzed

Table 21 - Stage 1 Analytical Results, All Processes

<u>Ion</u>	<u>Combined Reject (ug/l)</u>	<u>Microfilter Filtrate (ug/l)</u>	<u>WAC Effluent (ug/l)</u>	<u>RO Permeate (ug/l)</u>	<u>Boron Ion Exchange (ug/l)</u>		<u>Draft Monthly Average * (ug/l)</u>	<u>Report Only (ug/l)</u>
Antimony	0.62	<3	3.46	<0.6	<0.6		---	5
Arsenic	4.61	<3	<3	<0.6	<0.6		6	---
Barium	14.8	12.9	<10	<2	<2		---	7
Beryllium	< 0.10	<0.5	<0.5	<0.1	<0.1		---	0.25
Boron	5,850	5,770	5,690	253	<6		250	---
Cadmium	0.35	<1	<1	<0.2	<0.2		3	---
Calcium	13,900	16,800	340	250	280		---	---
Cobalt	19.0	<3	<3	<0.6	<0.6		---	46
Copper	4.30	<3	<3	<0.6	<0.6		10	---
Iron	< 10	58.0	<10	<10	<10		---	16
Lead	0.45	<1.5	<1.5	0.51	<0.3		---	2.5
Magnesium	8,420	2,370	<100	<100	<100		---	---
Manganese	27.1	<5	<5	2.60	<1		---	12
Mercury	7.3 ppt	2.0 ppt	2.9 ppt	2.5 ppt	na		2.1 ng/l	---
Molybdenum	9.10	9.52	9.21	<0.6	<0.6		---	5.5
Nickel	1,350	95.9	4.24	<0.3	6.18		---	24.5
Potassium	49,600	71,600	70,800	2,590	3,390		---	6,000
Selenium	20.0	17.8	17.5	<0.6	<0.6		5	---
Silver	< 6	<1	<1	<0.2	<0.2		0.4	---
Sodium	1,310,000	1,740,000	1,700,000	58,000	77,400		---	150,000
Strontium	1,610	1,740	7.84	<0.6	1.71		---	4.75
Thallium	10.0	9.63	6.18	<0.2	<0.2		---	2
Vanadium	< 1.0	<5	<5	<1	1.6		---	1.5
Zinc	31.5	16.3	<10	14.4	na		---	85
Ammonia -N	17,700	11,400	na	10,600	9,930		5,000	---
Chloride	1,390,000	1,960,000	na	69,600	136,000		---	220,000
Fluoride	< 100	< 100	na	< 100	<100		---	205
Sulfate	883,000	927,000	na	2,980	1,750		---	8,500
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Table 22 - Stage 2 Analytical Results, All Processes

Ion	Microfilter Feed (ug/l)	Microfilter Filtrate (ug/l)	WAC Effluent (ug/l)	RO Permeate (ug/l)	Boron Ion Exchange (ug/l)		Draft Monthly Average * (ug/l)	Report Only (ug/l)
Antimony	na	na	1.08	<0.6	<0.6		---	5
Arsenic	na	na	1.39	<0.6	<0.6		6	---
Barium	na	na	2.0	<2	<2		---	7
Beryllium	na	na	<0.1	<0.1	<0.1		---	0.25
Boron	na	na	14,300	624	<6		250	---
Cadmium	na	na	<0.2	<0.2	<0.2		3	---
Calcium	na	na	<100	670	510		---	---
Cobalt	na	na	<0.6	<0.6	<0.6		---	46
Copper	na	na	2.53	<0.6	<0.6		10	---
Iron	na	na	<10	18.0	<10		---	16
Lead	na	na	<0.3	<0.3	<0.3		---	2.5
Magnesium	na	na	<100	<100	<100		---	---
Manganese	na	na	<1	<1	<1		---	12
Mercury	420	na	14	3	1		2.1 ng/l	---
Molybdenum	na	na	10.70	<0.6	<0.6		---	5.5
Nickel	na	na	3.01	4	0.55		---	24.5
Potassium	na	na	56,600	2,520	1,950		---	6,000
Selenium	na	na	18.5	<0.6	<0.6		5	---
Silver	na	na	3.240	<0.2	<0.2		0.4	---
Sodium	na	na	2,400,000	75,100	87,900		---	150,000
Strontium	na	na	0.66	21	16.70		---	4.75
Thallium	na	na	3.49	<0.2	<0.2		---	2
Vanadium	na	na	<1	<1	<1		---	1.5
Zinc	na	na		14.4	14.4		---	85
Ammonia -N	na	na	na	6,850	7,570		5,000	---
Chloride	na	na	na	52,400	97,800		---	220,000
Fluoride	na	na	na	<100	<100		---	205
Sulfate	na	na	na	2,040	4,140		---	8,500
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Table 23 - Stage 3 Analytical Results, All Processes

Ion	Microfilter Feed (ug/l)	Microfilter Filtrate (ug/l)	WAC Effluent (ug/l)	RO Permeate (ug/l)	Boron Ion Exchange (ug/l)		Draft Monthly Average * (ug/l)	Report Only (ug/l)
Antimony	<3	<3	<3	<0.6	<0.6		---	5
Arsenic	4.22	<3	3.00	<0.6	<0.6		6	---
Barium	26.8	<10	<10	<2	<2		---	7
Beryllium	<0.5	<0.5	<0.5	<0.1	<0.1		---	0.25
Boron	17,100	15,900	16,500	7,120	1,160		250	---
Cadmium	<1	<1	<1	<0.2	<0.2		3	---
Calcium	38,400	14,900	190	210	280		---	---
Cobalt	8.7	<3	<3	<0.6	<0.6		---	46
Copper	5.96	3.68	<3	1.61	<0.6		10	---
Iron	87.000	<10	<10	<10	10.0		---	16
Lead	<1.5	2.06	<1.5	<0.3	0.32		---	2.5
Magnesium	14,900	120	<100	<100	<100		---	---
Manganese	9.9	<5	<5	<1	<1		---	12
Mercury	na	na	na	na	na		2.1 ng/l	---
Molybdenum	12.80	12.10	11.10	<0.6	<0.6		---	5.5
Nickel	653	6.2	2.86	<0.3	1.52		---	24.5
Potassium	78,000	72,700	67,500	2,280	2,100		---	6,000
Selenium	24.2	24.0	22.8	<0.6	<0.6		5	---
Silver	11.500	1.380	<1	<0.2	<0.2		0.4	---
Sodium	2,890,000	3,010,000	2,860,000	72,300	69,300		---	150,000
Strontium	4,940	3,490	<3	<0.6	1.18		---	4.75
Thallium	11.9	10.80	<1	<0.2	<0.2		---	2
Vanadium	<5	<5	<5	<1	<1		---	1.5
Zinc	na	na	na	na	na		---	85
Ammonia -N	11,000	10,500	na	5,420	4,870		5,000	---
Chloride	3,490,000	3,530,000	na	52,400	97,800		---	220,000
Fluoride	<100	<100	na	<100	<100		---	205
Sulfate	1,080,000	1,040,000	na	2,040	3,180		---	8,500
* Michigan Department of Environmental Quality Groundwater Discharge Permit - Draft Permit No.GW1810162								